

Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) EP 1 211 329 A2

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
05.06.2002 Bulletin 2002/23

(51) Int Cl.7: C21D 1/613, C21D 1/76,
C21D 1/74

(21) Application number: 01128749.7

(22) Date of filing: 03.12.2001

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE TR
Designated Extension States:
AL LT LV MK RO SI

(72) Inventors:
• Van den Syde, Jaak
Katonah, New York 10536 (US)
• Jaynes, Scot Eric
Lockport, New York 14094 (US)

(30) Priority: 04.12.2000 US 727473

(74) Representative: Schwan - Schwan - Schorer
Patentanwälte
European Patent Attorneys
Elfenstrasse 32
81739 München (DE)

(71) Applicant: PRAXAIR TECHNOLOGY, INC.
Danbury, CT 06810-5113 (US)

(54) Process and apparatus for high pressure gas quenching in an atmospheric furnace

(57) Apparatus and process for recycling a quenching gas, such as helium, to be used with a treating gas, such as a carburizing gas, for the treating of components in an atmospheric furnace.

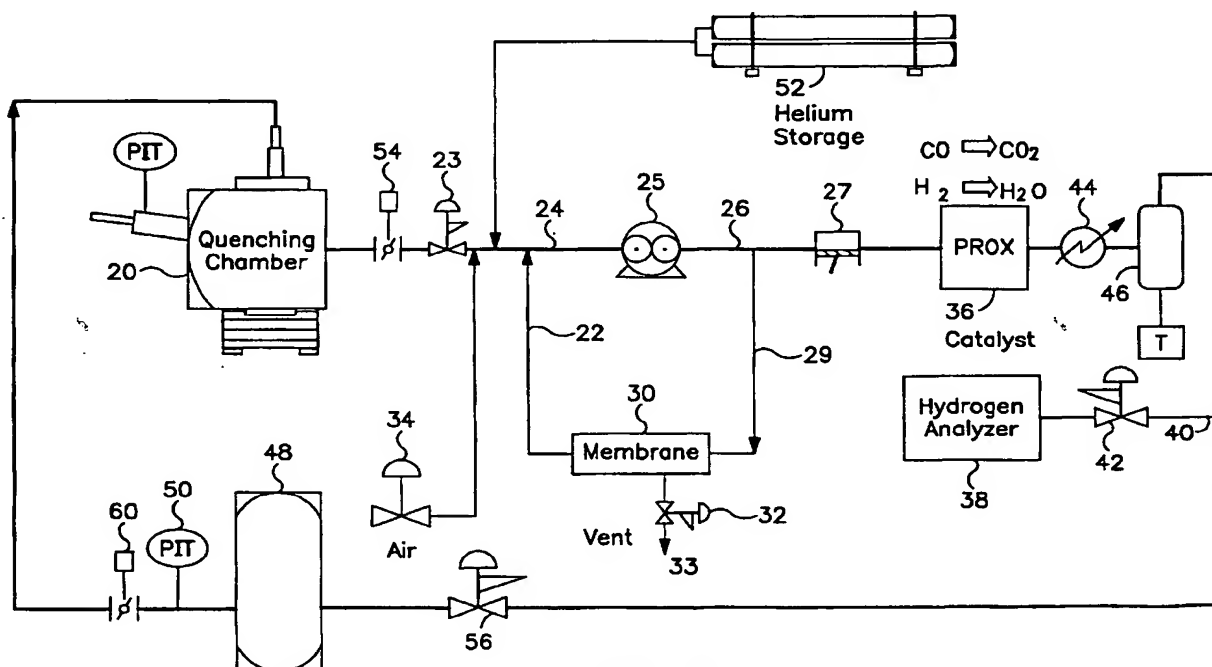


FIG. 3

DescriptionField of the Invention

5 [0001] The present invention is directed to a process and apparatus for recycling and purifying a quenching gas, such as helium gas, in the presence of a treating gas, such as carburizing gas, for use with an atmospheric furnace for treating components.

Background of the Invention

10 [0002] Conventionally, the hardening or treating of components, such as steel components, generally requires a heat treatment followed by a rapidly quenching treatment using a fluid such as oil. The process using oil can cause safety and environmental concerns. Exposing oil to a temperature of 900°C could cause the oil to volatilize and/or oxidize. The oxidized oil represents a degradation of the oil that must be filtered out of the quenching bath or removed by
 15 changing the oil. In either case, the oxidized oil and oil changes represent a waste stream that should be disposed of or partly recycled. Generally, oil remains on the treated components removed from the oil quench bath. Oil tends to drip off the components as they are handled and moved to the cleaning area. Fires, slips/falls and other hazards can occur as a result of using an oil quench process. Spent quenching-oil coated components may require an additional cleaning step before they are shipped or machined.

20 Additionally, quenching with oil may cause the components to distort significantly. To solve the problems posed by the use of oil as a quenching medium, gas, such as helium, has been used to cool components after they had been heated in a furnace.

[0003] United States Patent No. 5,158,625 discloses a process for heat treating articles by hardening them in a recirculating gas medium which is in contact with the treated articles. The hardening gas is cooled by means of a heat
 25 exchanger, of the type in which helium is used as hardening gas, and is stored under holding pressure in a buffer container. At the end of a hardening operation, a helium load is extracted from the treatment enclosure, in final phase by means of a pump until a primary vacuum is obtained. The extracted helium is brought to purifying pressure by means of a compressor associated to a mechanical filter, and the helium under pressure is sent to a purifier in which impurities are removed, after which it is transferred, if desired, after recompression in the buffer container.

30 [0004] United States Patent No. 5,938,866 discloses an apparatus for the treatment of components by means of a gas mixture, comprising mainly a first light gas and minor amounts of a second gas being heavier than the first gas. The apparatus has a treatment chamber, where the treatment occurs and a concentration, and purification device in which the gas mixture is concentrated and purified to increase the concentration of the first gas. The treatment chamber comprises an outlet member provided in an upper part of the treatment chamber and means being arranged to move
 35 the gas mixture upwardly and out through the outlet member.

[0005] United States Patent No. 4,867,808 discloses a process for heat treatment of metallic workpieces by heating in a vacuum furnace followed by quenching in a coolant gas under above-atmospheric pressure and with coolant-gas circulation.

40 [0006] United States Patent No. 5,173,524 discloses a rapid gas quenching process wherein an increased cooling rate of an article heated to an elevated temperature is achieved by flowing an inert gas mixture of helium and another inert gas over the article under conditions of turbulent flow.

[0007] It is an object of the present invention to provide a process for recycling a quenching gas in an atmospheric heat-treating furnace system.

45 [0008] It is another object of the present invention to provide a process for recycling a quenching gas, such as helium, in an atmospheric carburizing furnace system.

[0009] It is an object of the present invention to provide an apparatus for the gas treatment of components in an atmospheric furnace and having means for the quenching of the components from the atmospheric furnace using a recycling quenching gas.

50 [0010] It is another object of the present invention to provide a process and apparatus that effectively provides means for recycling a quenching gas, such as helium, in an atmospheric carburization furnace in which the quenching gas is maintained at a consistent quenching atmosphere as required for the component to be processed.

Summary of the Invention

55 [0011] The invention relates to a process for heat treating components in an atmospheric heat-treating furnace comprising the steps of:

(a) treating a component in an atmospheric furnace with a treating gas, such as an endothermic gas, heated to a

desired temperature required to treat the component;

(b) feeding the heat treated component containing the treating gas into a quenching chamber;

(c) feeding a quenching gas into the quenching chamber to contact the treated component and mix with the treating gas;

(d) feeding the quenching gas and treating gas mixture of step (c) into a gas recovery chamber where the treating gas and quenching gas are separated to provide a purified quenching gas and treating gas;

(e) feeding the purified quenching gas of step (d) back into the quenching chamber thereby effectively recycling the quenching gas back to the quenching chamber; and

(f) removing the cooled treated component from the gas quenching chamber.

[0012] If the furnace is approved for quenching pressure, then the quenching chamber could be eliminated and therefore reference to the quenching chamber in steps (b), (c), (e) and (f) shall mean furnace as recited in step (a) of the novel process of this invention.

[0013] The process of this invention is suitable for treatment of components manufactured from carbon, alloy and tool steels. Of particular importance are the carburizing grades of steel such as AISI grades 5120, 8115, 8620 and 9310. A primary use of the novel process of this invention is for use in atmospheric carburizing furnaces in which the treating gas can be at least one gas selected from the group comprising methane, methane, carbon monoxide, nitrogen, propane and butane. A common treating gas for carburizing is endothermic gas which consists of about 20% carbon monoxide, 40% hydrogen and 40% nitrogen. The treating gas could be heated to about 750°C and about 1200°C, preferably about 800°C and about 1000°C. For carburization treatment of components, the carburizing gas would be heated between about 850°C and about 1100°C, and preferably about 900°C and about 950°C. The quenching gas could be at least one gas selected from the group consisting of helium, preferably as the major component (>50%) and from the group consisting of nitrogen, argon and carbon monoxide as the minor component. The preferred quenching gas would be helium. The quenching gas should be pressurized at least to 37 psia and preferably between about 74 psia and about 890 psia, and more preferably between about 147 psia and about 368 psia. The quenched treated component is generally removed from the quenching chamber at atmospheric pressure and slightly above ambient temperature.

[0014] The subject invention also relates to an apparatus for the treatment of components by a gas in an atmospheric furnace comprising an atmospheric furnace adapted for receiving treating gas and a component to be gas treated, the atmospheric furnace coupled to a quenching chamber which is adapted for receiving the treated component from the atmospheric furnace and a quenching gas; the quenching chamber coupled to a gas recovery device adapted for receiving spent treating gas and quenching gas and having means for separating the gases to provide a purified quenching gas; the gas recovery device coupled to the quenching chamber and adapted for transmitting the purified gas into the quenching chamber; and the apparatus operable such that quenching gas can be recycled between the quenching chamber and the recovery device.

Brief Description of the Drawings

[0015] Figure 1 is a schematic of a gas quenching system.

[0016] Figure 2 is a schematic of a helium/endothermic gas quenching system of the present invention.

[0017] Figure 3 is a schematic of another embodiment of a helium gas quenching system of the present invention.

Detailed Description of the Invention

[0018] Figure 1 shows an equipment orientation that will allow helium quenching for an atmospheric carburizing furnace using an endothermic gas or a vacuum carburizing furnace using a gas such as propane or methane. At the end of the carburizing step, furnace 1 is opened and the components and furnace atmosphere enter, via duct 2, heated vacuum chamber 3. Heated vacuum chamber 3 is sealed from furnace 1 and helium quenching chamber 5. Upon closure of heated vacuum chamber 3, the atmosphere is removed via vacuum pump 9. During the entire process, heated vacuum chamber 3 remains at the furnace temperature so that the components do not start to cool. Following the removal of atmosphere from heated vacuum chamber 3 the chamber may or may not be back filled with helium at a pressure, for example, about 14.7 psia. The components will move to helium quenching chamber 5 when the chamber has met the following conditions. Chamber 5 is empty of the previous load of components, the chamber has been sealed from the outside atmosphere, and the outside atmosphere has been removed from chamber 5 via vacuum pump 9. Once the seal between chambers 3 and 5 is broken the components will move to chamber 5 and the seal established once again between chambers 3 and 5. Chamber 5 will then receive helium at the quenching pressure (e. g. 290 psia). Following the quenching operation the helium is removed from chamber 5 via duct 10 to helium recovery system 11 and then the components are moved to the next step in the process, for example, machining. The spent

helium is purified to a desired level in helium recovery system 11 and the purified helium is returned to chamber 5 via duct 12.

[0019] Atmospheric carburizing processes that quench with helium find the use of helium very expensive if they do not recycle the helium or require additional capital cost for the furnace equipment. The subject invention recycles helium with carburizing gases present and maintains a consistent quenching atmosphere as needed for the components being processed. Figure 2 shows one embodiment of a novel process of the subject invention. Several components disclosed in Figure 1 have the same numerical indicators as components in Figure 2. Carburized components plus the furnace atmosphere are moved directly to quenching chamber 3 and then sealed from furnace 1. Quenching chamber 3 is then pressurized with quenching gas from the quenching gas recovery system and the components are quenched. The quenching gas plus furnace atmosphere is then removed from quenching chamber 3 via quenching gas recovery system 7. The quenched components are then moved on to the next step in the process (e.g. machining 5).

[0020] Figure 2 shows the difference between the embodiment described by Figure 1 above and the subject invention. Subject invention results in reduced equipment cost and process complexity. Both require the use of a quenching chamber. However, previous recycle systems were not feasible to remove the carburizing gases.

[0021] Table 1 shows a typical carburization gas composition that would enter the quenching chamber when an evacuation of the chamber is not performed. In addition to water a significant amount of carbon dioxide, carbon monoxide, methane, hydrogen and nitrogen enter into the system. With the addition of the quenching gas to 20 bar, the carburization gas will represent 5% of the total gas in the quenching chamber.

TABLE 1

Compound	% of Carburization Gas	Mass in Quenching Chamber (lbs)
CO ₂	1	0.08
CH ₄	1	0.03
H ₂ O	2	0.07
CO	19	0.98
H ₂	38	0.28
N ₂	39	2.02

[0022] Purifying the spent quenching gas would not be possible without oxygen since oxygen must be added to remove hydrogen. The subject invention could use a catalyst followed by a molecular sieve to purify the entire quenching gas stream and return pure helium to the quenching chamber.

[0023] Figure 3 shows a gas recovery system in which quenching gas flows from quenching chamber 20 via duct 24 to the suction side of oil flooded screw compressor 25. The pressure of the suction side of oil flooded screw compressor 25 is controlled to a maximum by pressure regulator 23. Oil flooded screw compressor 25 will discharge the quenching gas at 150 psig or higher. The discharge of oil flooded screw compressor 25 will pass through oil removal equipment (not shown) in duct 26 and then through the suction side of diaphragm compressor 27. The discharge of compressor 27 is at a higher pressure such as 575 psig (~40 bar absolute)

[0024] Between the oil removal equipment and compressor 27, approximately 60% of the total flow through diaphragm compressor 27 of the quenching gas will take side branch 29 and pass through membrane 30. The membrane will discard methane, carbon monoxide, carbon dioxide and nitrogen through valve 32. The membrane permeate will return to the suction side of oil flooded screw compressor 25 via duct 22 and duct 24. The stream compositions for the feed, permeate and raffinate are given below in Table 2. The membrane feed as shown in Table 2 represents the steady state composition of the gas in the quenching chamber (i.e. quenching gas plus endo gas). The permeate at approximately 94% pure helium will mix with the unpurified gas and pass through the catalyst and water removal. The composition of gas in the receiver before equalization with the quenching chamber is approximately 95% pure helium. Upon equalization with the quenching chamber, the endo gases as shown in Table 1, lowers the helium purity to approximately 90%.

Oxygen was not shown in the simulation below but would be present because air inlet valve 34 feeds the suction of compressor 25. Oxygen is completely consumed in the conversion of hydrogen to water and carbon monoxide to carbon dioxide. The presence of oxygen in the membrane is expected to have an insignificant impact on the helium recovery and final steady state gas composition.

TABLE 2

CALCULATED PROCESS PARAMETERS For Membrane 19			
	FEED	RAFT	PERM
F,MMSCFD (60F)	1	0.0504	0.9496
PRESS, psia	150.00	150.00	6.00
TEMP, F	108.00	108.00	108.00
Molec. Weight	6.35	27.06	5.26
Viscos, cp	0.0205	0.0182	0.0204
CONCENTRATIONS, Mol%			
HELIUM	89.8000	10.0000	94.0354
NITROGEN	3.8000	61.3748	0.7442
HYDROGEN	2.1000	0.4062	2.1899
CARBON MONOXIDE	1.0000	15.6039	0.2249
WATER	0.2000	0.0008	0.2106
CARBON DIOXIDE	3.0000	10.9495	2.5781
METHANE	0.1000	1.6648	0.0169

Percent recovery of helium in stream No. 22 = 99.44

Percent recovery of nitrogen in stream No. 33 = 81.40

Percent recovery of hydrogen in stream No. 33 = 0.97

Percent recovery of carbon monoxide in stream No. 33 = 78.64

Percent recovery of water in stream No. 33 = 0.02

Percent recovery of carbon dioxide in stream No. 33 = 18.40

Percent recovery of methane in stream No. 33 = 83.91

[0025] The hot gas from diaphragm compressor 27 passes through catalyst bed 36 to convert some of the hydrogen to water and carbon monoxide to carbon dioxide. Oxygen is provided for the reaction by air inlet valve 34 at the suction side of oil flooded screw compressor 25. Valve 34 allows the air to enter the quenching gas recovery system and is controlled by a signal from hydrogen analyzer 38. When the level of hydrogen is over a predetermined set point, hydrogen analyzer 38 will send a signal to valve 34 to let in air. Analyzer 38 maintains an excess of hydrogen in the system. The combination of catalyst and excess hydrogen will cause the removal of oxygen to the PPM level such as <10 PPM. The hydrogen analyzer is located in duct 40 after valve 42. Following catalyst 36, the gas stream is cooled in heat exchanger 44 and passed through separator 46 to remove entrained water. The entrained water passes to a trap and is discharged from the system. The trap may operate by a float or a timer (T). The trap seals the quenching gas recovery system from outside air and does not allow quenching gas to escape from the quenching gas recovery system. The quenching gas will fill quenching gas ballast tank 48 from valve 56 until the pressure reaches, for example, 590 psig as measured by PIT 50. Not all of the gas in the quenching chamber is removed to the quenching gas recovery system and some quenching gas is lost during purification with membrane 30. Replacing the lost quenching gas with helium is done at the suction side of oil flooded screw compressor 25. When the suction pressure of oil flooded screw compressor 25 falls below a predetermined set point, then make up helium will flow from helium storage 52 through a control valve (not shown).

[0026] Once quenching gas ballast tank 48 reaches a predetermined set point pressure, then the quenching gas recovery system has finished and shuts down. When the quenching gas recovery system shuts down, butterfly valve 54 closes. Air/nitrogen or other gas back fills the quenching chamber and the components are removed. The empty chamber is closed and purged with nitrogen or other gas. A new load of hot components is then placed in quenching chamber 20 and quenching gas ballast tank 48 is equalized with quenching chamber 20 through butterfly valve 60. The next cycle begins.

[0027] For the preferred embodiment of the subject invention, quenching gas pressure requirements of approximately 10 bar or less would use only one compressor. For a helium purity of approximately 90% and carbon monoxide con-

version, the compressor could circulate 60% of the recovered gas in the quenching chamber through the compressor and through the membrane. Therefore, the compressor could remove 875. CF of quenching gas from the quenching chamber. From the discharge of the compressor, 525 CF could pass through the membrane back to the suction side of the compressor. For a cycle time of 15 minutes, the compressor would move 1400 CF or 5600 SCFH. Thus, compressor 27 is significantly smaller at 3500 SCFH. A smaller compressor 27 saves on capital cost and operating cost over the prior art. A water separator could be used to remove entrained water (Figure 3 #46). Heater exchanger 44 could be augmented with a chiller for lower volumes of water in the quenching gas. The amount of water in the quenching gas should remain constant as a saturated gas, at the temperature and pressure of the stream, entering ballast tank 48. [0028] Modifications can be made to the quenching gas recovery system of the subject invention as follows:

1. Quenching chamber 20 would not be required if the furnace chamber is approved for quenching pressures. The quenching gas recovery system would remain the same.
 2. A separate vacuum pump could be used in a side process connected to duct 24 before valve 23 to evacuate quenching chamber 20 so that a greater percentage of quenching gas is recovered. The vacuum pump would be turned on after the quenching chamber reached atmospheric pressure.
 3. The oil flooded screw and diaphragm compressors could be replaced with other style compressors and/or combined into one compressor.
 4. The purification side stream that flows through the membrane could take place anywhere after the discharge of compressor 27.
 5. Purification of the side stream could replace membrane 30 with molecular sieve or a purge.
 6. Purification of the side stream could use molecular sieve or another membrane on the raffinate stream of membrane 30 to increase helium recovery. Also, the raffinate could be placed in a separate receiver and serve as purge gas for the quenching chamber.
 7. Molecular sieve or another membrane could be added to the permeate of membrane 30 for additional purification.
 8. Valve 34 could inlet pure oxygen instead of air.
 9. Heat exchanger 44 could be augmented with a chiller to further reduce the amount of water in the quenching gas.
 10. The system could run continuously if a line and valve were placed between the quenching gas ballast tank 48 and the suction side of oil flooded screw compressor 25, thus, allowing the system to run continuously would increase the helium content of the quenching gas or a smaller compressor 25 to get the same helium content in the quenching gas.
 11. More than one quenching chamber can be used in one quenching gas recovery system. Equipment can be sized based on the number of quenching chambers and the controls are adjusted so that the quenching gas in each quenching chamber can reach the desired gas composition and pressure.
 12. Minimizing the amount of oxygen present in quenching chamber 20 during the quenching step would require a purge gas through the quenching chamber before the introduction of hot components. The purge gas could be nitrogen, argon, helium or used endothermic gas from the carburizing process.
 13. To accomplish an oxygen free quenching chamber, a separate chamber could be added that receives the components from the quenching chamber. The additional chamber could have a purge of nitrogen, argon, or helium.
 14. Ballast tank 48 could provide purge gas to quenching chamber 20. The quenching gas recovery system would be set up to run continuously. However, after the components are removed from quenching chamber 20, valves 60 and 54 would open and allow gas to purge quenching chamber 20 for a period of time. At the end of the purge, valve 56 would close first and then valve 22 would close, leaving the chamber at near atmospheric pressure. Then the next cycle would start with the addition of hot components to quenching chamber 20.
 15. The flow of gas through duct 29 could be reduced resulting in lower helium purity as the quenching gas. Helium purity of 40% or greater may be used depending on the desired cooling curves in the quenching chamber.
 16. Oxygen or air can be introduced to the quenching gas recovery system after compressor 27. Introduction of additional gas after the compressor would reduce the flow through valve 34 since the membrane would discard none of the oxygen. This option would have the most value when pure oxygen was being used to oxidize hydrogen and carbon dioxide.
 17. Catalyst temperature and type can be adjusted to minimize or practically eliminate the conversion of carbon monoxide to carbon dioxide. To maintain a helium purity of 90% requires only 40% of the stream through duct 29 when carbon monoxide is not oxidized.
- A 40% flow in duct 29 represents a 33% decrease over the preferred method as described above. Table 3 shows the feed, raffinate and permeate compositions when carbon monoxide is not converted to carbon dioxide. The membrane is approximately four times as efficient at discharging carbon monoxide as it is carbon dioxide. Another advantage is that less oxygen consumption is required for quenching gas recovery system oxidation. This option would be the preferred method if reduction in the quenching chamber of carbon dioxide to carbon

monoxide is possible and undesirable.

TABLE 3

CALCULATED PROCESS PARAMETERS For Membrane 30 Without CO Conversion			
	FEED	RAFT	PERM
F,MMSCFD (60F)	1	0.07797	0.922
PRESS, psia	150.00	150.00	6.00
TEMP, F	108.00	108.00	108.00
Molec. weight	6.18	25.55	4.54
Viscos, cp	0.0206	0.0185	0.0203
CONCENTRATIONS, Mol%			
HELIUM	88.8000	10.0000	95.4633
NITROGEN	5.6000	58.9144	1.0917
HYDROGEN	2.1000	0.4003	2.2437
CARBON MONOXIDE	2.8000	28.5080	0.6261
WATER	0.2000	0.0007	0.2169
CARBON DIOXIDE	0.4000	1.0937	0.3413
METHANE	0.1000	1.0829	0.0169

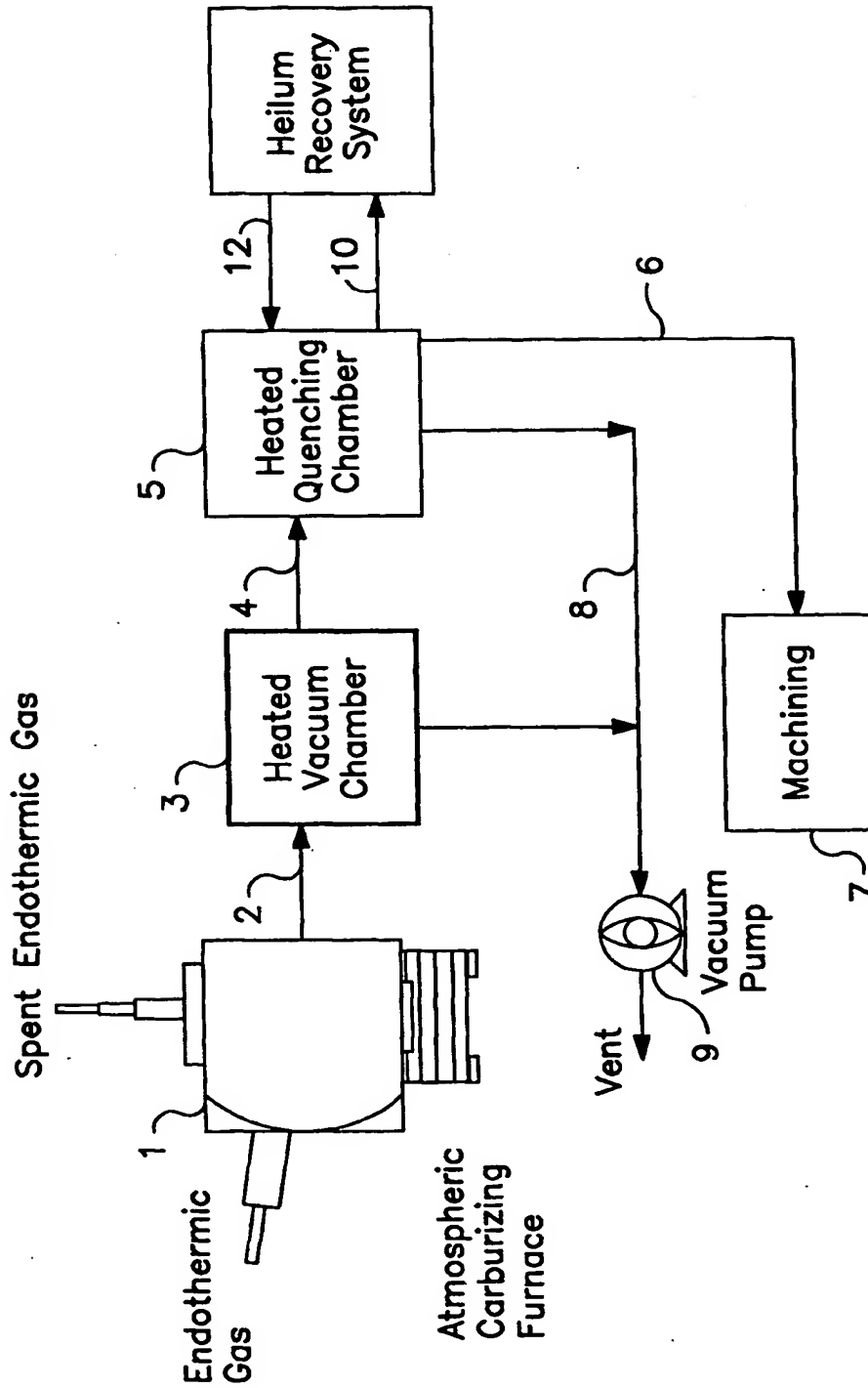
Percent recovery of helium in stream No. 22 = 99.12
 Percent recovery of nitrogen in stream No. 33 = 82.02
 Percent recovery of hydrogen in stream No. 33 = 1.49
 Percent recovery of carbon monoxide in stream No. 33 = 79.38
 Percent recovery of water in stream No. 33 = 0.03
 Percent recovery of carbon dioxide in stream No. 33 = 31.32
 Percent recovery of methane in stream No. 33 = 84.43

[0029] The invention is not limited to the embodiment shown and it will be appreciated that it is intended to cover all modifications and equipment within the scope of the appended claims.

Claims

1. A process for heat treating components in an atmospheric heat treating furnace comprising the steps:
 - (a) treating a component in an atmospheric furnace with a treating gas;
 - (b) feeding the heat treated component containing the treating gas into a quenching chamber;
 - (c) feeding a quenching gas into the quenching chamber to contact the treated component and mix with the treating gas;
 - (d) feeding the quenching gas and treating gas of step (c) into a gas recovery chamber where the treating gas and quenching gas are separated to provide a purified quenching gas;
 - (e) feeding the purified quenching gas of step (d) back into the quenching chamber; and
 - (f) removing the cooled treated component from the gas quenching chamber.
2. The process of claim 1 wherein the atmospheric furnace is a carburizing atmospheric furnace and the treating gas is selected from the group comprising methane, carbon monoxide, hydrogen, nitrogen, pentane and butane.
3. The process of claim 1 wherein the quenching gas is at least one gas selected from the group comprising helium as the major component and one gas selected from the group comprising nitrogen, hydrogen, argon and carbon dioxide.

4. The process of claim 1 wherein the treating gas is heated to a temperature between about 750°C and about 1200°C.
5. The process of claim 1 wherein the quenching gas is pressurized to a pressure between about 37 psia and about 890 psia.
- 5 6. An apparatus for the treatment of components by a gas in a furnace comprising a furnace adapted for receiving treating gas and a component to be gas treated; said furnace coupled to a quenching chamber which is adapted for receiving the treated component from the furnace and the quenching gas; said quenching chamber coupled to a gas recovery device adapted for receiving the spent treating gas and the quenching gas and having means for separating the gases to provide a purified quenching gas; said gas recovery device adapted for transmitting the purified gas into the quenching chamber; and said apparatus operable such that quenching gas can be recycled between the quenching chamber and the recovery device.
- 10 7. The apparatus of claim 6 wherein the furnace is an atmospheric furnace.
- 15 8. The apparatus of claim 6 wherein the gas recovery device comprises a membrane adapted for purifying the quenching gas.
- 20 9. The apparatus of claim 8 wherein the gas recovery device comprises a molecular sieve.
- 25 10. The apparatus of claim 8 wherein hydrogen analyzing means are coupled to the recovery device for monitoring the hydrogen in the purified gas; oxygen feed means are coupled to the quenching chamber; and control means are coupled between said hydrogen analyzing means and oxygen feed means for controlling the feed of oxygen depending on the analysis of the hydrogen in the purified gas.



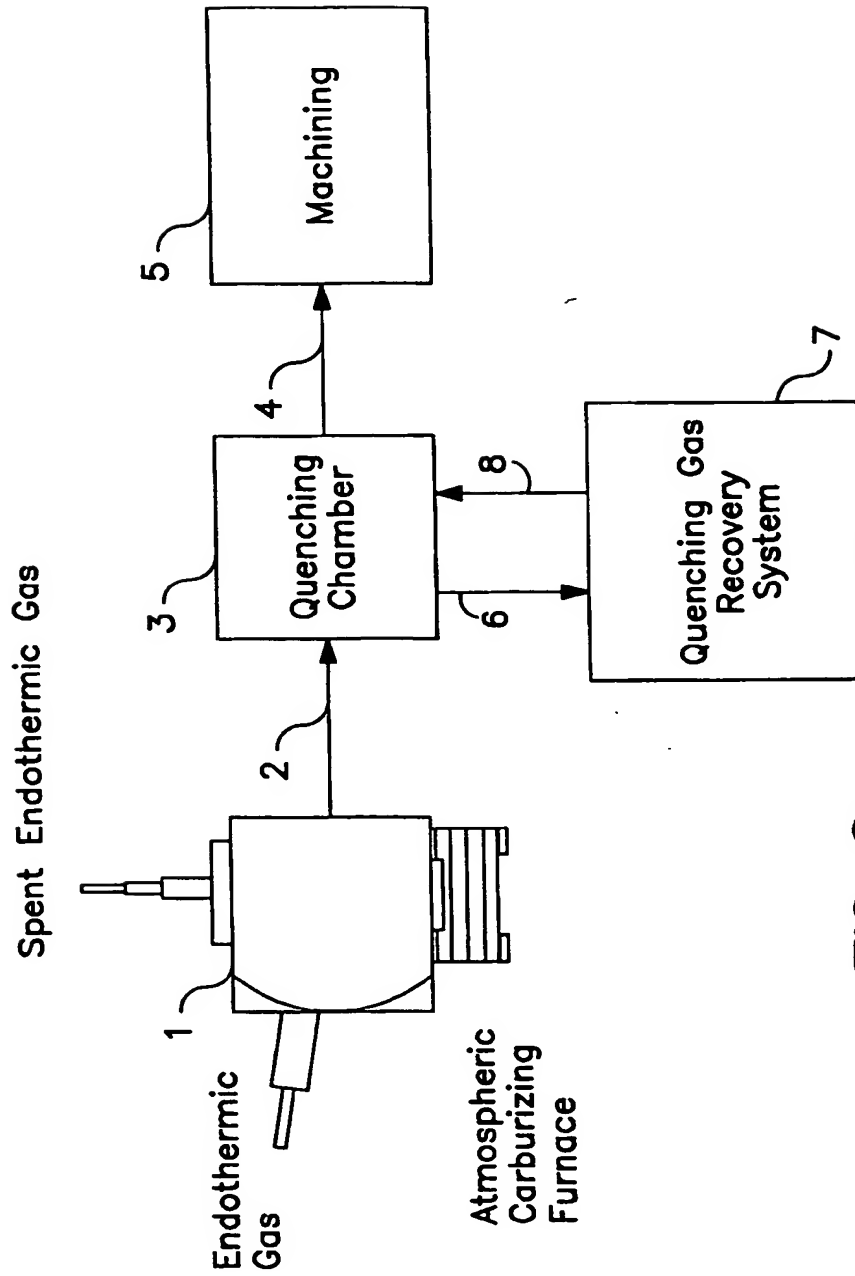


FIG. 2

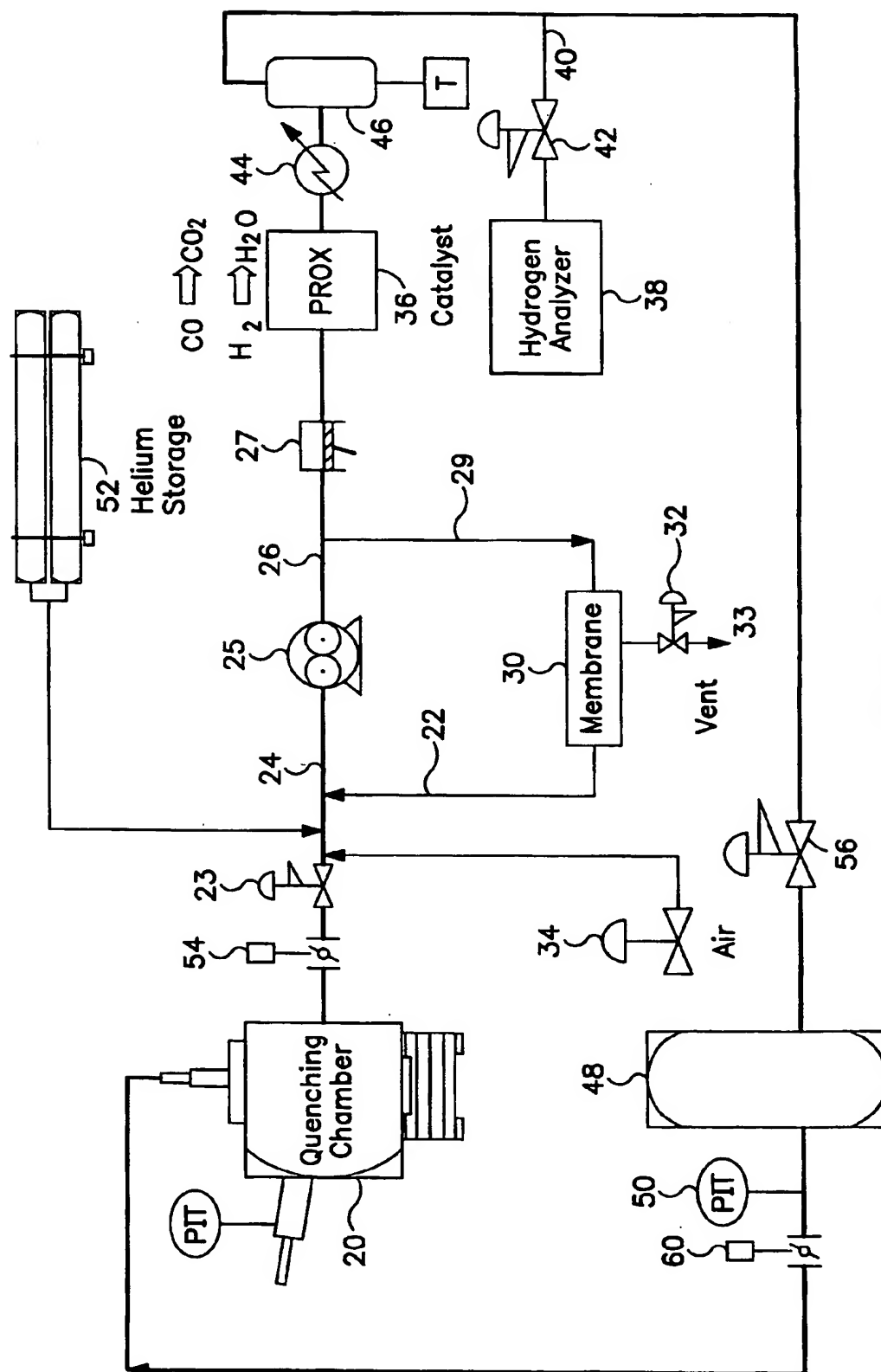


FIG. 3

This Page Blank (uspto)